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Ultrasonic, volumetric and viscometric study of molecular interactions in binary mixtures of 2,2,4-trimethyl pentane with *n*-hexane and cyclohexane at 308 K

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Abstract Experimental values of density, ultrasonic speed and viscosity for the binary mixtures of 2,2,4-trimethyl pentane with n-hexane and cyclohexane are presented at 308 K over the entire mole fraction range From these data, isentropic compressibility, relative association, acoustic impedance, molar sound speed, molecular association, deviations in isentropic compressibility, excess volume, deviations in ultrasonic speed, excess acoustic impedance and deviations in viscosity have been calculated These parameters were used to study the nature and extent of intermolecular interactions between component molecules in the present binary mixtures. The ultrasonic speeds and viscosities were theoretically evaluated using different empirical relations and theories The relative merits of these relations and theories were discussed

Keywords Ultrasonic speed, viscosity, liquid mixtures

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1. Introduction

The study of molecular interactions in binary liquid mixtures has attracted the attention of scientific community for more than a century; the study finds direct applications in chemical and biological industry. Mixed solvents rather than single pure liquids are of utmost practical importance in most chemical and industrial processes as they provide a wide range of mixtures of two or more components in varying proportions so as to permit continuous adjustment of desired properties of the medium. Several experimental studies aimed at finding experimental evidence for intermolecular interactions in binary liquid mixtures have been carried out by us [1–3]. As an extension to this study, we focus in this paper the results of binary mixtures of 2,2,4-trimethyl pentane (TMP) with n-hexane and cyclohexane. All the three components are non-polar, unassociated, inert hydrocarbons [4] and cyclohexane possesses globular structure [5]. Alkanes have been studied extensively in view of their importance in petrochemical industries, particularly in the light of the present day trends towards heavier feedstock [6, 7]. But an exhaustive survey of literature indicates that no work has been done on these systems from the point of view of their ultrasonic, volumetric and viscometric behaviour.

The densities ρ, ultrasonic speeds u, and viscosities η, of pure TMP, n-hexane, cyclohexane and those of their binary mixtures with TMP as common component have been measured, covering the entire composition range, at 308 K. From these experimental data, isentropic compressibility K_s, relative association R_A, acoustic impedance Z, molar sound speed R_m, molecular association M_A, deviations in isentropic compressibility ΔK_s, excess volume V^E, deviations in ultrasonic speed Δu, excess acoustic impedance Z^E and deviations in viscosity $\Delta\eta$, have been calculated. The dependence of these parameters on composition of the mixture reveals the nature and extent of interaction between component molecules. Ultrasonic speed was theoretically evaluated by using free length theory (FLT) [8], Nomoto's relation [9] and van Deal and Vangeel's relation [10].

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The relative merits of these theories and relations were examined. The viscosity data were used to test the semi-empirical relations of Grunberg and Nissan [11], Tamura and Kurata [12], Hind *et al* [13], Katti and Chaudhri [14] and Heric [15] by comparing the theoretical values of viscosity obtained from these relations with the experimental values.

2. Experimental

TMP, n-hexane and cyclohexane (all s.d. fine, A.R. grade) were purified by standard methods described in the literature [16]. The mixtures were prepared by weight and were kept in special airtight bottles. The weighings were done on an Afcoset-ER-120A electronic balance with a precision of 0.1 mg. The probable error in mole fraction was estimated to be less than ±0.0001.

The densities of pure liquids and binary mixtures were measured using a single capillary pycnometer (made of Borosil glass) of bulb capacity 8.0×10^{-3} dm^3. The capillary with graduated marks had uniform bore and could be closed using a well-fitting glass cap. The marks on the capillary were calibrated with triple distilled water. The accuracy in density measurements was found to be ±0.0001 g cm^{-3}. The ultrasonic speeds were measured using a single-crystal variable path ultrasonic interferometer (Mittal Enterprise, New Delhi, Model-M-82) at 3 MHz with an accuracy of ±0.5%. Viscosities were measured using Ubbelohde viscometer calibrated with triple distilled water. The viscometer containing the test liquid was allowed to stand for 20 minutes in thermostatic bath so that thermal fluctuation in the viscometer was minimised. The accuracy in viscosity measurements was found to be $\pm 3 \times 10^{-6}$ Nm^{-2} s. The temperature of the test liquids and their binary mixtures was maintained at 308 ± 0.05 K in an electronically controlled thermostatic water bath.

3. Results and discussion

The experimentally measured values of ρ, u and η of pure liquids and their binary mixtures as a function of mole fraction x of TMP at 308 K are listed in Table 1. From the measured values of ρ, u and η, K_s, R_A, Z, R_m and M_A have been calculated using the following standard relations [1,3] :

$$K_s = u^{-2}\rho^{-1} \quad (1)$$

$$R_A = (\rho/\rho_0)\,(u_0/u)^{1/3} \quad (2)$$

$$Z = u\,\rho \quad (3)$$

$$R_m = u^{1/3}V \quad (4)$$

$$M_A = [(u^2/u_{im}^2) - 1] \quad (5)$$

where ρ_0, ρ, and u_0, u are the densities and ultrasonic speeds of solvent and solution, respectively. V is the molar volume of the mixture and u_{im} is the ultrasonic speed of ideal mixture. The values of u_{im} were obtained using the method of van Dael and Vangeel [10]. The variations of K_s, R_A, Z, R_m and M_A with mole fraction x of TMP are presented in

Table 1. Values of density (ρ), ultrasonic speed (u), and viscosity (η), for the binary mixtures at 308 K

x (TMP)	ρ (kg m^{-3})	u (ms^{-1})	$\eta(10^{-3}$ N m^{-2} s)
	TMP + *n*-hexane		
0.0000	646.0	1401.6	0.2588
0.0810	648.8	1038 1	0 2666
0 1655	651 8	1036 0	0 2764
0 2537	655 1	1036 2	0.2870
0.3458	658.4	1037.1	0 2988
0 4423	661.8	1040.0	0.3117
0 5433	665.2	1043.0	0 3257
0 6492	668.6	1047.1	0.3416
0 7603	672.0	1048.1	0.3589
0 8771	675 6	1048.0	0 3783
1 0000	679.2	1045 4	0 4017
	TMP + cyclohexane		
0 0000	764 4	1211 1	0 6920
0.0679	756.3	1195 2	0 6489
0 1407	747.9	1176.8	0 6076
0 2192	739 4	1160.7	0 5706
0 3040	730 8	1143 4	0 5383
0.3958	722.2	1128.0	0 5045
0.4956	713.6	1112.8	0 4761
0.6045	705 0	1096.0	0 4515
0.7238	696 4	1079 5	0 4290
0 8550	687 8	1060 5	0 4081
1.0000	679.2	1045 4	0 4017

Table 2. It is observed from Table 2 that the values of K_s decrease and those of R_A and Z increase for the system TMP + n-hexane, while a reverse trend in these parameters is observed for the system TMP + cyclohexane. The values of R_m increase in both the systems under study. The observed increase or decrease in K_s, R_A, Z, R_m and M_A with composition of the mixture is an evidence of the presence of interaction between the components of the binary liquid mixtures under study. Similar conclusion was also arrived at by others [17,18].

The deviations in isentropic compressibility ΔK_s, excess volume V^E, deviations in ultrasonic speed Δu, excess acoustic impedance Z^E and deviations in viscosity $\Delta\eta$, have been calculated from the relation :

$$Y^E = Y_{mix} - [(1 - x)\,Y_1 + xY_2], \quad (6)$$

where x is the mole fraction of TMP (volume fraction ϕ is used in case of ΔK_s). Y^E is K_s or V^E or Δu or Z^E or $\Delta\eta$ and

Table 2. Calculated values of isentropic compressibility (K_s), relative association (R_A), specific acoustic impedance (Z), molar sound speed (R_m) and molecular association M_A for the binary mixtures at 308 K.

x (TMP)	K_s (10^{-9} $m^2 N^{-1}$)	R_A	Z (10^5 kg $m^{-2} s^{-1}$)	R_m [10^{-3} $m^3 mol^{-1}$ $(m s^{-1})^{1/3}$]	M_A
			TMP + *n*-hexane		
0.0000	1.4268	1.0000	6.7287	1.3523	0.0000
0.0810	1.4302	1.0055	6.7352	1.3804	−0.0012
0 1655	1.4294	1.0108	6.7526	1.4099	−0 0007
0 2537	1.4217	1.0158	6.7881	1.4411	0 0032
0.3458	1.4121	1.0207	6.8283	1.4741	0.0073
0 4423	1.3970	1.0250	6.8827	1.5093	0.0139
0 5433	1.3819	1.0293	6.9380	1.5462	0.0192
0.6492	1.3641	1.0332	7.0009	1 5855	0.0247
0 7603	1.3546	1.0381	7.0432	1 6251	0.0221
0 8771	1 3477	1.0437	7.0803	1.6656	0 0148
1 0000	1.3472	1.0501	7 1004	1.7069	0.0000
			TMP + cyclohexane		
0 0000	0.8919	1 0000	9 2576	1 1736	0.0000
0 0679	0 9256	0.9938	9 0393	1.2096	−0.0032
0 1407	0.9655	0 9878	8.8013	1.2478	−0 0099
0 2192	1.0039	0 9811	8.5822	1 2899	−0 0120
0 3040	1.0467	0.9746	8.3560	1.3350	−0 0152
0 3958	1.0882	0.9674	8 1464	1.3846	−0 0142
0 4956	1.1316	0.9603	7.9409	1.4386	−0 0117
0 6045	1.1808	0.9535	7.7268	1.4966	−0 0109
0 7238	1.2322	0.9467	7.5176	1 5603	−0 0081
0.8550	1.2928	0.9405	7.2941	1.6290	−0 0086
1 0000	1.3472	0.9332	7 1004	1 7069	0.0000

subscripts 1 and 2 stand for n-hexane/cyclohexane and TMP, respectively. The values of K_s, V^E, Δu, Z^E and $\Delta\eta$ for each mixture have been fitted to the polynomial equation :

$$Y^E = x(1-x)\sum_{i=1}^{j} A_i(1-2x)^{i-1}. \quad (7)$$

The values of the coefficients A_i calculated by the method of least squares along with the standard deviations $\sigma(Y^E)$ are listed in Table 3. The coefficients A_i are the adjustable parameters for a better fit of the excess functions.

The sign and magnitude of ΔK_s and V^E play a vital role in assessing the molecular rearrangement as a result of molecular interactions between the component molecules in the liquid mixtures. In general, negative values of ΔK_s and V^E indicate strong interactions in the liquid mixtures which include charge-transfer, dipole-dipole, dipole-induced-dipole interactions and interstitial accommodation of the smaller molecules into the spaces created by the bigger molecules, while positive signs of these parameters are indicative of the weakening of interaction between the component molecules [18]. Figures 1 and 2 show that for TMP + *n*-hexane binary mixtures, ΔK_s shows positive deviations up to ~0.58 mole fraction of TMP, whereas V^E values are entirely positive over the whole composition range. Positive deviations in ΔK_s and V^E may be attributed to the presence of weak interaction between the component molecules in the mixture. Similar trends in ΔK_s and V^E are also observed in 2-butoxy ethanol + toluene [19] and alkyl acetate + *n*-hexane [20] binary mixtures, respectively. For TMP + cyclohexane binary mixtures, both ΔK_s and V^E are entirely negative over the whole composition range. Since all the three components of the present binary mixtures are non-polar and inert solvents, strong intermolecular interactions between the component

Table 3. Coefficients A_i of eq. (7) along with standard deviations $\sigma(Y^E)$ for the binary mixtures at 308 K.

Function	A_1	A_2	A_3	A_4	A_5	$\sigma(Y^E)$
			TMP + *n*-hexane			
ΔK_s(10^{-10} $m^2 N^{-1}$)	0 2337	1 7707	−0 2073	−0.6665	0 7413	0.0068
V^E(10^{-7} $m^3 mol^{-1}$)	6.8194	2 7583	7.5906	3.7383	−3.5915	0.0539
Δu($m s^{-1}$)	−7 0663	−65.3464	−13.8999	27.0447	−31.2706	0.2454
Z^E(10^3 kg $m^{-2} s^{-1}$)	0 0400	−43 9512	6.8122	16.2704	−19.6795	0.1747
$\Delta\eta$(10^{-2} N m^{-2} s)	−4.2468	0.3793	−0.1499	0 1623	−2.0474	0.0099
			TMP + cyclohexane			
ΔK_s(10^{-10} $m^2 N^{-1}$)	−1 3302	−0 1288	0.8515	−0 8387	−0 8427	0 0098
V^E(10^{-7} $m^3 mol^{-1}$)	−2.5887	−2 3740	−2.0417	−3.2093	−3.2127	0.0144
Δu($m s^{-1}$)	−64 5941	−27.9555	−58.1139	35 9524	58 8808	0.5377
Z^E(10^3 kg $m^{-2} s^{-1}$)	−98.7185	−32.549	−44.9682	27.8917	48.2144	0 4080
$\Delta\eta$(10^{-2} N m^{-2} s)	−28.3828	−7.6667	−4.2429	6 8380	−5.0206	0.0735

molecules are not feasible. The only factor which seems to be primarily responsible for the observed positive values of ΔK_s and V^E for TMP + *n*-hexane and negative values for

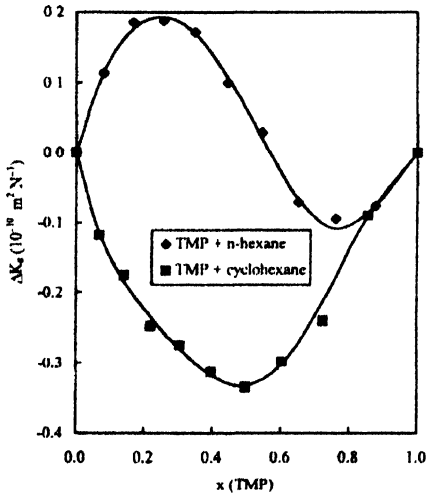

Figure 1. Plots of deviations in isentropic compressibility ΔK_s against mole fraction x of TMP for the binary mixtures at 308 K (■/◆ experimental points; —— calculated curves).

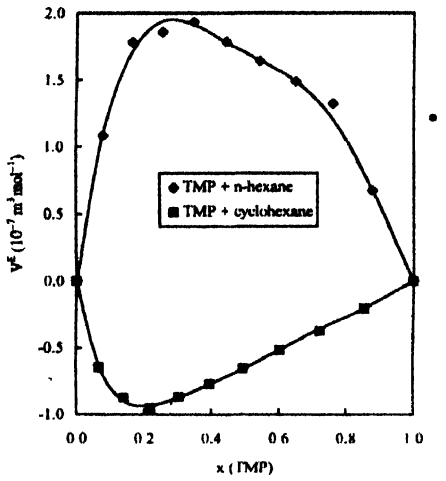

Figure 2. Plots of excess volume V^E against mole fraction x of TMP for the binary mixtures at 308 K (■/◆ experimental points; —— calculated curves).

TMP + cyclohexane systems is the interstitial fitting of the component molecules into each other's structure. Larger molar volume difference between TMP (molar volume = 1.68×10^{-4} m^3 mol^{-1}) and cyclohexane (molar volume = 1.10×10^{-4} m^3 mol^{-1}) than that between TMP and n-hexane (molar volume = 1.33×10^{-4} m^3 mol^{-1}) would allow a more favourable packing of cyclohexane molecules than n-hexane molecules into the spaces created by TMP molecules. This would result in a contraction in volume, hence, negative ΔK_s and V^E values for the system TMP + cyclohexane, and an expansion in volume, leading to positive ΔK_s and V^E values for the system TMP + *n*-hexane. This is in accordance with the view proposed by Fort and Moore [18], according to which liquids of too different molecular size usually mix with decrease in volume yielding negative ΔK_s and V^E values. However, in his recent study of alkanes, Patterson [21] reported that there is orientational order between longer n-alkane (as *n*-hexane in the present study) molecules, which is destroyed when such alkanes are mixed with other destroyer *e.g.* TMP, resulting in positive ΔK_s and V^E values Negative trends in ΔK_s and V^E have also been reported for ternary liquid mixtures of cyclohexane + decane + butanol [5].

As expected, Figures 3 and 4 show that the behaviours of Δu and Z^E support each other. Both exhibit negative deviations over the entire composition range except for small positive deviations in case of TMP + *n*-hexane, at higher mole fractions of TMP. Negative deviations in Δu and Z^E from rectilinear dependence on composition suggest that

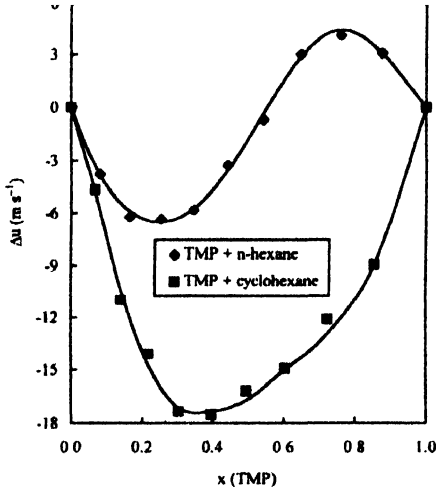

Figure 3. Plots of deviations in ultrasonic speed Δu against mole fraction x of TMP for the binary mixtures at 308 K (■/◆ experimental points; —— calculated curves)

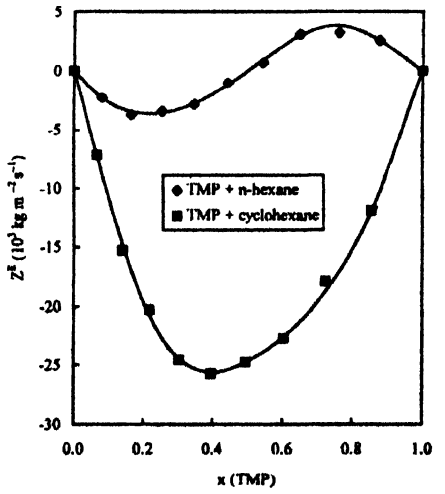

Figure 4. Plots of excess acoustic impedance Z^E against mole fraction x of TMP for the binary mixtures at 308 K (■/◆ experimental points; —— calculated curves).

dispersion forces are operative in these systems. This seems in good agreement with the conclusion arrived at by Fort and Moore [18,22] for the study of excess functions for binary liquid mixtures. Negative deviations in Δu and Z^E have also been reported for binary mixtures of cyclohexane + benzene/ CCl_4 [23].

The graphical presentation of experimental $\Delta\eta$ plotted against the mole fraction x of TMP is shown in Figure 5.

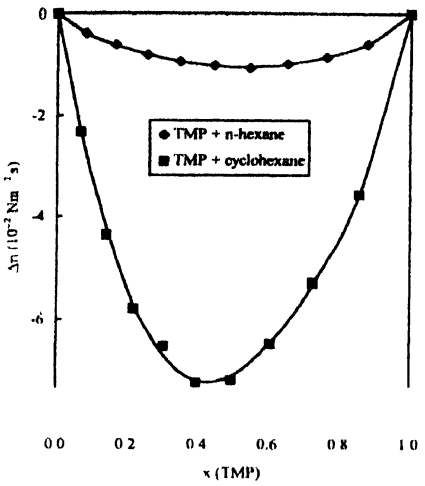

Figure 5. Plots of viscosity deviations $\Delta\eta$ against mole fraction x of TMP for the binary mixtures at 308 K (■/◆ experimental points, —— calculated curves)

Positive deviations from ideal behaviour indicate strong interaction between unlike molecules while negative deviations point to the presence of dispersion forces. The observed negative values of $\Delta\eta$ over the whole composition range for both the binary systems investigated (Figure 5) suggest that dispersion forces prevail between the component molecules. This further reinforces our view discussed earlier. Such trends in the variation of $\Delta\eta$ with composition are also reported for benzene + cyclohexane/ethylene dichloride binary mixtures [22].

Theoretical evaluation of ultrasonic speed in liquid mixtures offers a simple and convenient method for the study of the nature of molecular interaction in these mixtures. The theoretical values of ultrasonic speed in the systems under study were evaluated using the following relations :

Free length theory (FLT) [8] :

$$u\,(\mathrm{FLT}) = K/(L_f \rho^{1/2}), \tag{8}$$

Nomoto's relation (NOM) [9] :

$$u(\mathrm{NOM}) = \{[(1-x)R_1 + xR_2]/[(1-x)V_1 + xV_2]\}, \tag{9}$$

van Dael and Vangeel's ideal mixing relation (VD) [10] :

$$1/u^2_{i,a} = [(1/M_1u_1^2) + (1/M_2u_2^2)][(1-x)M_1 + xM_2]. \tag{10}$$

The details of derivations and terms used in these relations are given in the literature [8–10,24,25]. The calculated ultrasonic speeds along with the experimental speeds and percentage error in the calculated values from those of the experimental values are summarised in Table 4. On comparison, the results indicate that for TMP + n-hexane mixtures FLT provides good agreement between theoretical and experimental ultrasonic speeds followed by Nomoto's relation, while van Dael and Vangeel's relation shows maximum deviation. For TMP + cyclohexane binary mixtures, Nomoto's relation seems to provide best results followed by van Dael and Vangeel's relation and then by FLT. However, we are working to develop a better equation for reproducing the experimental values of ultrasonic speed, thus, to develop a better liquid structure model.

Table 4. Theoretical values of ultrasonic speed calculated from NOM, FLT and VDV relations along with experimental ultrasonic speed and percentage error at 308 K.

x	u (m s^{-1})				% Error		
(TMP)	Expt	FLT	NOM	UDV	FLT	NOM	UDV
			TMP + n-hexane				
0 0000	1041 6	1041.6	1041 6	1041 6	0 00	0 00	0 00
0 0810	1038 1	1040 0	1042 0	1038 7	0 18	0.37	0.06
0 1655	1036.0	1039.1	1042.4	1036 4	0.30	0.61	0.04
0 2537	1036.2	1039.4	1042 7	1034.5	0 31	0 63	0 16
0 3458	1037.1	1039.7	1043.1	1033.3	0 25	0.58	0.36
0 4423	1040.0	1040.4	1043 5	1032.8	0 04	0 34	0.69
0.5433	1043.0	1041.1	1043.9	1033 2	0 18	0.08	0 94
0.6492	1047.1	1041.8	1044 3	1034 4	0.51	0.27	1 21
0 7603	1048.1	1042.5	1044.6	1036.7	0 53	0.33	1.09
0.8771	1048.0	1044.0	1045 0	1040.3	0.39	0.28	0.73
1.0000	1045.4	1045.4	1045 4	1045 4	0.00	0 00	0.00

Table 4. *(Cont'd.)*.

x (TMP)	u (m s^{-1})				% Error		
	Expt	FLT	NOM	UDV	FLT	NOM	UDV
			TMP + cyclohexane				
0.0000	1211.1	1211.1	1211.1	1211.1	0.00	0 00	0.00
0.0679	1195.2	1189.2	1193.8	1197.1	0.51	0.12	0.16
0.1407	1176.8	1167.6	1176.6	1182.7	0.78	0.01	0.50
0.2192	1160.7	1147.6	1159.7	1167.7	1.13	0.09	0.60
0.3040	1143.4	1128 8	1142.8	1152.2	1.28	0.05	0.77
0.3958	1128.0	1111.6	1126.2	0136.1	1.45	0.16	0.72
0.4956	1112.8	1095 9	1109.7	1119.4	1.51	0 28	0.59
0.6045	1096.0	1081.6	1093.4	1102.0	1.31	0.24	0.55
0 7238	1079 5	1068.5	1077.2	1083 9	1.02	0.21	0.41
0.8550	1060.5	1056.4	1061.2	1065.1	0.38	0 07	0.43
1.0000	1045.4	1045.4	1045.4	1045 4	0.00	0.00	0.00

Table 5. Values of the parameters of eqs. (11)–(15) and corresponding standard percentage deviations in correlating viscosities of the binary mixtures at 308 K.

Binary mixture	eq (11)		eq. (12)		eq. (13)		eq. (14)		eq (15)	
	G_{12}	σ(%)	C	σ(%)	η_{12}	σ(%)	W_{vis}/RT	σ(%)	Δ_{12}	σ(%)
TMP + n-hexane	–0.0445	0.16	2.5955	17.70	0.3077	0.16	–0.0115	0 14	–0.0046	0 15
TMP + cyclohexane	–0.3633	0.46	4.8587	28.54	0.3997	1.41	–0.2751	0.40	–0.3159	0.43

The theoretical values of viscosity in the mixtures : TMP + *n*-hexane and TMP + cyclohexane were calculated using the following semi-empirical relations :

Equation of Grunberg and Nissan [11]

$$\eta = \exp\,(x_1 \ln\,\eta_1 + x_2 \ln\,\eta_2 + x_1 x_2 G_{12}), \qquad (11)$$

where G_{12} is the interaction parameter.

Equation of Tamura and Kurata [12]

$$\eta = x_1 V_1 \eta_1 + x_2 V_2 \eta_2 + 2(x_1 x_2 V_1 V_2)^{1/2} C, \qquad (12)$$

where V_i is the molar volume of pure component i and C is an adjustable parameter.

Hind *et al* [13] proposed the following equation :

$$\eta = x_1^2 \eta_1 + x_2^2 \eta_2 + 2x_1 x_2 \eta_{12}, \qquad (13)$$

where η_{12} is the interaction parameter.

Katti and Chaudhri [14] derived the following equation :

$$\ln \eta V = x_1 \ln V_1 \eta_1 + x_2 \ln V_2 \eta_2 + x_1 x_2 W_{vis}/RT, \qquad (14)$$

where W_{vis} is an interaction term.

The Heric's [15] equation is of the form :

$$\ln \eta = x_1 \ln \eta_1 + x_2 \ln \eta_2 + x_1 \ln M_1 + x_2 \ln M_2 + \ln(x_1 M_1 + x_2 M_2) + \Delta_{12}, \qquad (15)$$

where Δ_{12} (= $\alpha_{12} x_1 x_2$) is a function representing molecular interaction and $\alpha_{12} = \alpha_{21}$ is the interaction parameter. Heric expressed α_{12} or α_{21} as a linear function of composition. The values of interaction parameters calculated from the above equations together with the standard percentage deviations are presented in Table 5. Comparing the results given in Table 5 we conclude that for both the binary systems the relations proposed by Grunberg and Nissan [11], Hind *at al* [13], Katti and Chaudhri [14] and Heric [15] reproduce the experimental data well, showing small percentage deviations, while the relation proposed by Tamura and Kurata [12] gives large deviations.

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